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DEVELOPMENT OF A MODEL AND COMPUTER CODE TO DESCRIBE SOLAR GRADE SILICON PRODUCTION PROCESSES

SEVENTH QUARTERLY REPORT

R. SRIVASTAVA AND R.K. GOULD

AUGUST 1979



The JPL Low-Cost Solar Array Project is sponsored by the U. S. Department of Energy and forms part of the Solar Photovoltaic Conversion Program to initiate a major effort toward the development of low-cost solar arrays. This work was performed for the Jet Propulsion Laboratory, California Institute of Technology by agreement between NASA and DoE.

AeroChem Research Laboratories, Inc.
Princeton, New Jersey

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Approved by


Hartwell F. Calcote
Director of Research

AeroChem Research Laboratories, Inc.
Princeton, New Jersey

ABSTRACT

This program aims at developing mathematical models and computer codes based on these models, which allow prediction of the product distribution in chemical reactors for converting gaseous silicon compounds to condensed-phase silicon. The major interest is in collecting silicon as a liquid on the reactor walls and other collection surfaces. Two reactor systems are of major interest, the Westinghouse SiCl₄/Na reactor in which Si(l) is collected on the flow tube reactor walls and the new AeroChem reactor in which Si(l) droplets formed by the SiCl₄/Na reaction are collected by a jet impingement method.

During this quarter the following tasks have been accomplished: (i) particle deposition routines have been added to the boundary layer code and (ii) Si droplet sizes in SiCl₄/Na reactors at temperatures below the dew point of Si are being calculated. Initial results from the latter show that droplet sizes in such reactors are predominantly in the range 0.2 to 1.0 μm . Such particles are too small to collect by conventional means (e.g., cyclones) but should be easily collected by very high velocity impaction at reduced pressures such as that being examined by AeroChem.

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I. INTRODUCTION

In this report we consider the introduction of particles into the existing model¹ for the vapor collection efficiency of silicon reactors of the Westinghouse type.² The last report¹ concerned the partial collection onto the reactor walls of silicon vapor present in the reactor's hot core flow (see Fig. 1 for a schematic of the physical situation). While the deposition of vapor was attributed to the processes of convection, Fick diffusion, and turbulent (or eddy) transport to the reactor walls, the deposition of particles (silicon droplets) is expected to be controlled by additional mechanisms. In particular, one expects Brownian diffusion to govern the deposition of small particles (10^{-6} - 10^{-2} μm diam) while Soret (or thermophoretic) transport will become more important for larger particles (10^{-2} - 1.0 μm diam), especially within the "viscous sublayer" region next to the reactor walls, where large temperature gradients exist. Away from the walls the particles will be transported entirely by diffusive turbulent velocity fluctuations. This latter effect will always tend to drive the particles close to the walls. As was shown earlier,^{3,4} near the walls the former two molecular mechanisms (i.e., Brownian and Soret effects) will predominate.

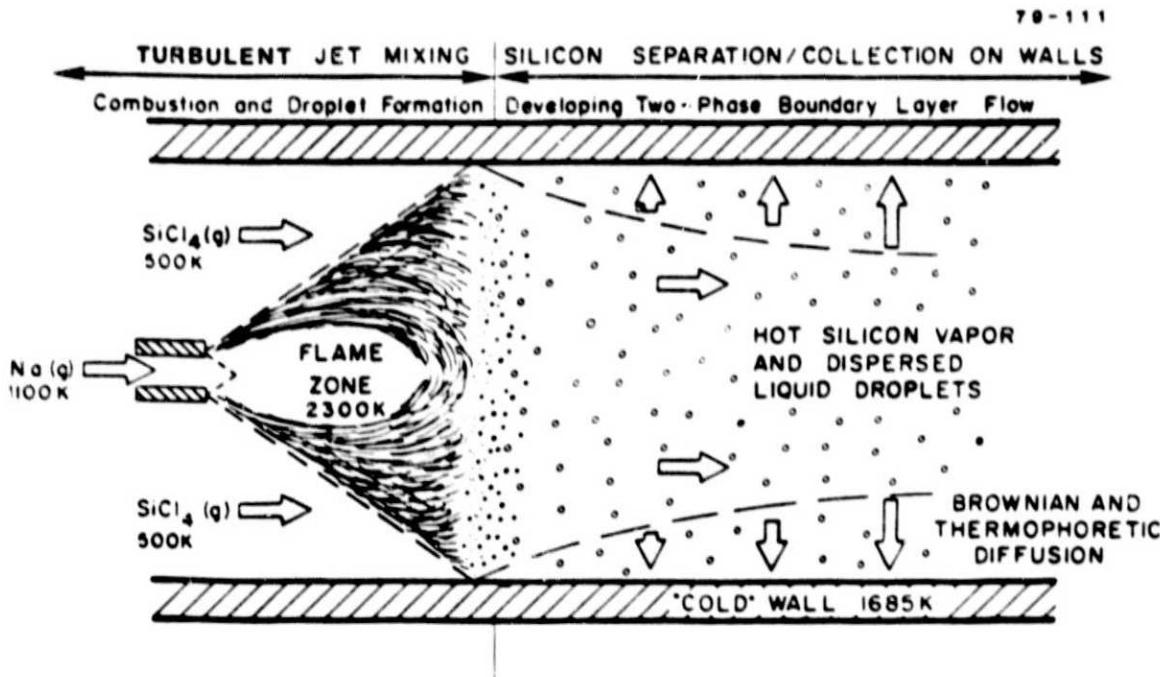


FIGURE 1 SCHEMATIC OF THE MODEL CONSIDERED

In order to describe the overall silicon collection efficiency (due to vapor and droplets) of a reactor, it is essential to model the deposition behavior of a distribution of particles' varying in size from molecular dimensions to about a micron diameter. In the model considered here it is assumed that the particle size distribution function (PSDF) for the silicon droplets entering the downstream section of the reactor for separation/collection is known.* The question, as before, is, "What fraction of the silicon vapor and droplets entering the reactor is collected on the walls?" The model described here attempts to answer this question, with due regard to the various deposition mechanisms mentioned earlier and the two-dimensional developing boundary layer flow within the reactor. In fact, as a result of the slowly developing boundary layers within typical reactors most of the deposition occurs prior to the establishment of "fully-developed" conditions (i.e., with no streamwise variations). Hence, the available simpler solutions which are valid for fully-developed, or partially developed (Graetz type)* flow cannot be used since such analyses only provide an asymptotic limit for the silicon mass transfer rate to the walls. Over the reactor's separation/collection length, the velocity, temperature, and concentration boundary layers are developing simultaneously. Hence, numerical solution of the coupled system of boundary layer conservation equations is unavoidable in determining the cumulative silicon collection efficiency, which depends upon the variation of the silicon mass flux (at the wall) along the length of the reactor.

This report outlines the general approach being adopted in the numerical modeling of the silicon separation/collection processes. Specific complications are isolated in order to show the nature of the problem and the methods used to overcome the difficulties encountered. The actual solutions for the collection efficiency will be presented in a subsequent quarterly report, which will discuss the model in greater detail.

The results of calculations using the modified LAPP code are also discussed in this report. Particle size calculations using this code to describe a reactor similar to the Westinghouse reactor, but operating at temperatures below the Si dew point, show that the silicon droplets produced rapidly grow to a medium size $\approx 0.4 \mu\text{m}$ in diameter.

* Actually, this PSDF will only be known after an analysis of the turbulent jet mixing, combustion, and nucleation processes in the upstream reactor section. This is, in fact, the main goal of the modified LAPP code being developed under this contract.

II. SILICON DROPLET DEPOSITION MODEL

A. PHYSICAL AND MATHEMATICAL ASPECTS

The present two-phase flow model for describing the transport of silicon particles from the hot (≈ 3500 K) core-flow of the reactor to the cooled walls (≈ 1685 K) is based on the assumptions that: (i) the total particle volume is considerably smaller than the volume of the surrounding gas phase so that particle-particle interaction is negligible; (ii) the concentration of the particles is small enough so that their presence does not alter the surrounding gas flow field (i.e., velocity and temperature distributions); (iii) the particles are small enough that they may be treated as "heavy-molecules" in local equilibrium with the gas.

These assumptions are usually justified in the downstream section of the silicon reactor being considered. The diluteness of the silicon droplets can be expected to increase with increasing distance from the droplet source, immediately following combustion (see Fig. 1). On the other hand, the nature of the processes leading to silicon droplet formation (gas-to-particle conversion via nucleation) ensures that the resulting particles will be of sub-micron sizes, in the absence of appreciable coagulation.

Under these assumptions, one can write the following particle mass conservation equation suitable to the developing, boundary layer flow within the reactor (axisymmetric):

$$\rho u \frac{\partial Y_i}{\partial x} + \rho v \frac{\partial Y_i}{\partial r} = - \frac{1}{r} \frac{\partial}{\partial r} [r j_{ir}] \quad (1)$$

where the radial mass flux is given by

$$j_{ir} \cong j_{ilaminar} + j_{iturbulent} \quad (2)$$

$$= - \rho D_i \left[\frac{\partial Y_i}{\partial r} + \alpha_i Y_i (1 - Y_i) \frac{1}{T} \frac{\partial T}{\partial r} \right] - \rho \epsilon_p \frac{\partial Y_i}{\partial r} \quad (3)$$

Expressions for the Brownian diffusivity, D_i , and the dimensionless Soret factor, α_i , were presented earlier.^{3,4} The turbulent diffusivity, ϵ_p , is taken to be unaffected by the small particles being considered and hence given by the

expressions suggested by Lin et al.* Equation (1) is valid provided the particles do not grow appreciably (via vapor collection on their surfaces) during passage through the particle diffusion layer. Such an assumption may be justifiable in view of the typical thinness of these layers, as seen later. Moreover, Eq. (1) applies to particles of a uniform size, present in the external stream, depositing by convection, Brownian diffusion, and thermophoresis on the reactor walls. In order to describe the deposition of particle size distributions, therefore, one needs to repeatedly solve Eq. (1) with the appropriate boundary condition on concentration at the diffusion layer's edge, corresponding to each particle size class. That is, a PSDF expressing the variation of particle concentration vs. size in the external stream is discretized into a finite number of independent size classes, with particles in a given class being characterized by uniquely defined transport coefficients, α_i and D_i . The velocity and temperature fields within the turbulent boundary layers on the reactor walls have to be supplied to the particle diffusion equation. In the present case, for this purpose, the entire velocity and temperature profile history along the reactor tube length is independently computed using a modification of the GENMIX code^{8,9} for the finite-difference solution of coupled momentum and energy equations (including variable fluid properties and the damping of turbulence near the reactor walls).

Even in the absence of turbulence, the effects of thermophoresis and Brownian diffusion can immensely complicate the analysis of silicon droplet deposition behavior within the reactor. Physically, one expects thermophoretic effects to be important in regions of the reactor flow field where temperature gradients are large (i.e., near the walls). On the other hand, the larger particle size class, for which thermophoretic deposition is significant, corresponds to Brownian diffusivities that are extremely small. There exists, therefore, a thin sublayer region next to the walls where both Brownian diffusion and thermophoresis may be important for particle deposition. Outside this sublayer, however, temperature gradients continue to remain strong so that thermophoresis remains important, and diffusion by Brownian motion of particles becomes negligible. Thus one can consider the presence of an "inner layer."

* This implies that the particles follow the turbulent eddy motions of the gas. Actually, particles near 1 μm diam might break away from the eddies due to their even inertia. Such inertial effects are excluded in the present diffusional model.

next to the walls, where thermophoretic transport of particles balances convection and Brownian diffusion. External to this is an "outer layer" wherein convective transport balances thermophoretic effects in conserving particle mass.* Although Eq. (1) is, in principle, general enough to describe both these layers simultaneously, from the standpoint of computational efficiency it is useful to consider the abovementioned division into different layers (due to their disparate thicknesses).

It has been established that, in analyzing the effects of thermophoresis, it is convenient to visualize the introduction into the boundary layer of additional pseudo "sink" and "suction" behavior.¹² The former will tend to deplete particle concentrations above the reactor walls while the latter acts towards increasing them. The net result of these opposing effects of thermophoresis is to appreciably increase the collection rate for the larger particles.¹³

B. COMMENTS ON THE NUMERICAL SOLUTION PROCEDURE

A few difficulties arise with the numerical solution of the particle transport problem, described in the previous section. They are briefly discussed in this section, together with strategies adopted to overcome them.

1. The Stiffness Problem

When the characteristic time scales associated with convection, Brownian diffusion, and thermophoresis in Eq. (1) become widely disparate, one is likely to encounter "stiffness" during the numerical solution process. The problem is manifested by unstable numerical solutions if the integration does proceed or by a failure of the integrator to march forward, along the reactor length. The former corresponds to a build-up of truncation error as the solution proceeds (often seen as a wildly fluctuating solution) while the

* It is to be noted that these layers are not related to the "viscous sublayer" region next to the walls where turbulent dissipation of eddies occurs. In fact, both the "inner" and "outer" thermophoretic layers may be embedded within the "viscous sublayer" region. Moreover, the division into "layers" adopted here is different from the asymptotic three-layer model implied by Refs. 10 and 11.

latter shows up as an inability of the integrator to meet the specified local error tolerance.

To overcome this problem, a "stiffness solver," based on Gear's variable order, variable step-size, integration formulae is adopted.¹⁴ A method of lines approach is utilized, suitably discretizing the derivatives along the radial coordinate, in order to solve Eq. (1) as an initial-value, ordinary differential equation. The "solver" employs an implicit scheme for greater stability and to allow marching in larger step sizes. Both these features are crucial to coping with the stiffness problem.

2. The Scaling Problem

Due to the disparate thicknesses of the "inner" and "outer" thermo-phoretic layers, discussed earlier, one runs into resolution difficulties if the adopted radial grid spacing approaches the thickness of the inner layer, or exceeds it.

In order to deal with this problem the following transformation of the radial coordinate is defined:

$$\tilde{r} = \frac{r_w - r}{r_w - r_m} \quad (4)$$

where r , r_w , and r_m are the radial locations corresponding to the point of interest, the reactor wall, and the edge of the particle diffusion layer. Note that the transformation restricts the domain of integration of Eq. (1) to the range $0 \leq \tilde{r} \leq 1$, regardless of particle size. It is, of course, necessary to specify the approximate thickness of the diffusion layer (i.e., $\delta_m = r_w - r_m$). Since the thickness of the thermal boundary layer (i.e., δ_T), at a given x , is known via prior solution of the energy equation, one may readily estimate δ_m using the known Lewis number, for that particle size class, from the relation*:

$$\delta_m \cong \delta_T (\text{Le}_g)^{1/3} \quad (5)$$

* A more cumbersome approach could have been to estimate δ_m by solving the boundary layer concentration integral equation.

3. Determination of the Variable Edge Boundary Condition

At the above value of δ_m a concentration boundary condition needs to be specified. While the other boundary condition on Eq. (1) is simply specified at the wall as $Y_i = 0$, the one at the "inner layer" edge must be obtained via a degenerate form of Eq. (1). As discussed earlier, the degenerate equation used here ignores the diffusion term and further assumes that the transverse convection of particles in the outer layer is negligible compared to their axial convection. Thus one can write the "outer layer" equation as

$$u \frac{dY_i}{dx} = K Y_i \quad (6)$$

where¹²:

$$K \equiv \left[\frac{1}{r} \alpha_i D_i + \frac{1}{\rho} \frac{\partial}{\partial r} (\rho D_i \alpha_i) \right] \frac{1}{T} \frac{\partial T}{\partial r} + D_i \alpha_i \frac{1}{T} \left\{ \frac{\partial^2 T}{\partial r^2} - \frac{1}{T} \left(\frac{\partial T}{\partial r} \right)^2 \right\} \quad (7)$$

and analytically solve it to obtain the following expression for the concentration at the outer edge of the "inner layer":

$$Y_i = Y_{i_0} \exp \left[\int_{x_0}^x \frac{K}{u} dx' \right] \quad (8)$$

where the subscript 'o' refers to some specified initial conditions (e.g., at the inlet to the silicon separation/collection section of the reactor).* Equation (8) emphasizes that the particle trajectories immediately outside the "inner layer" may be nearly parallel to the reactor walls. This is to be expected when the opposing thermophoretic and hydrodynamic forces acting on the particle nearly balance each other.

* A more exact alternative approach to modeling the outer layer will include the transverse convection term in Eq. (6) and, thus, require numerical solution of differential equations for the concentration along particle trajectories.

III. SILICON DROPLET GROWTH RATES

The modified Lemp code (now called CHEMPART) has been used to compute Si droplet formation and growth rates in an SiCl_4/Na reactor. The basic geometry is similar to that employed by Westinghouse,² i.e., a 15 cm diam tube. Na vapor at 1400 K is input as a jet of 7.5 cm diam with an outer flow of $\text{SiCl}_4/\text{H}_2/\text{H}/\text{Ar}$ at 3700 K. Input flows and conditions are summarized in Table I. The mixing in the model flow is undoubtedly slower than in the actual Westinghouse system since in the model SiCl_4 injection is tested as axial whereas in the actual reactor, SiCl_4 injection is transverse to the Na flow. A more serious discrepancy may be that, to date, we have not input sufficient enthalpy to maintain the outer flow temperature at > 300 K. H and H_2 immediately react

TABLE I
 SiCl_4 REACTOR CALCULATIONS: INITIAL CONDITIONS

	<u>Inner Flow^a</u>	<u>Outer Flow^a</u>
Velocity, cm s^{-1}	3550	1642
Temperature, K	1400	3700
Concentrations, mole fractions:		
SiCl_4	0.0	0.381
Na	0.8	0.0
H_2	0.15	0.429
H	0.0	0.095
Ar	0.05	0.095

^a The inner flow is assumed uniform for the transverse distance $r = 0$ to 3.75 cm. The outer flow is uniform from $r = 3.75$ to 7.5 cm. Reactor radius = 7.5 cm, pressure = 1.0 atm, and the SiCl_4 to Na input flow ratio is stoichiometric.

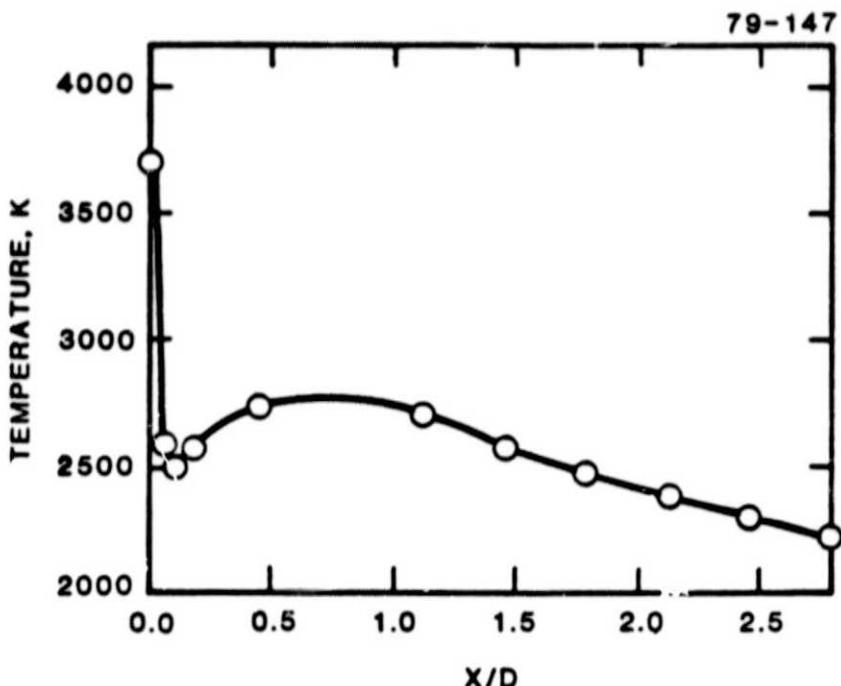
with SiCl_4 to form HCl and SiCl_2 predominantly via the reaction scheme described in Table II. In doing so the temperature rapidly drops to 2500 K. Equilibrium calculations show that, for the reactants and conditions of Table I, adiabatic reaction to equilibrium in the outer flow results in a temperature of 2350 K. Complete reaction with the Na in the inner flow leads to an adiabatic flame temperature of 2930 K. In future runs the enthalpy will be increased by increasing the H/H_2 input ratio, and mixing of the jet and outer flow will be enhanced

to more closely approximate the actual reactor conditions. (A final flame temperature of ≈ 3200 K will be sought.)

TABLE II
REACTION MECHANISM

1. Na + SiCl ₄	\rightleftharpoons	NaCl + SiCl ₃	14. H + SiCl ₄	\rightleftharpoons	HCl + SiCl ₃
2. Na + SiCl ₃	\rightleftharpoons	NaCl + SiCl ₂	15. H + SiCl ₃	\rightleftharpoons	HCl + SiCl ₂
3. Na + SiCl ₂	\rightleftharpoons	NaCl + SiCl	16. H + SiCl ₂	\rightleftharpoons	HCl + SiCl
4. Na + SiCl	\rightleftharpoons	NaCl + Si	17. H + SiCl	\rightleftharpoons	HCl + Si
5. Si + Si + M	\rightleftharpoons	Si ₂ + M	18. Si ₃ + Si ₃ + M	\rightleftharpoons	Si ₆ + M
6. Si + Si ₂ + M	\rightleftharpoons	Si ₃ + M	19. Si ₆ + Si ₆	\rightleftharpoons	Si ₁₂
7. Cl + Cl + M	\rightleftharpoons	Cl ₂ + M	20. Si ₁₂ + Si ₁₂	\rightleftharpoons	Si ₂₄
8. Na + HCl	\rightleftharpoons	NaCl + H	21. Si ₂₄ + Si ₂₄	\rightleftharpoons	Si ₄₈
9. Na + Cl ₂	\rightleftharpoons	NaCl + Cl	22. Si ₄₈ + Si ₄₈	\rightleftharpoons	Si ₉₆
10. H + HCl	\rightleftharpoons	H ₂ + Cl	23. Si ₉₆ + Si ₉₆	\rightleftharpoons	Si ₁₉₂
11. H + Cl ₂	\rightleftharpoons	HCl + Cl	24. Si ₁₉₂ + Si ₁₉₂	\rightleftharpoons	Si ₃₈₄
12. H + Cl + M	\rightleftharpoons	HCl + M	25. Si ₃₈₄	\rightleftharpoons	smallest particles
13. H + H + M	\rightleftharpoons	H ₂ + M			

At the lower (2500 K) temperatures now being calculated, Si particles form rapidly and it is of interest to describe particle growth in such cases (even though, in the condensation mode employed by Westinghouse, it is envisioned that they will operate above the Si dew point, i.e., at core flow temperatures well above 3200 K). In Fig. 2 the temperature, and in Fig. 3, the Si atom mole fraction and Si droplet mass loading are plotted vs. flow time. The radial position at which these plotted data were calculated is just beyond the edge of the inner flow, where reaction is initially the most intense. In Fig. 2 it is seen that in a very short distance, Reactions (7) and (12-17) of Table II result in a rapid temperature drop. The major species in the outer flow immediately become HCl and SiCl₃. As Na mixes into the region being described, the temperature rises to ≈ 2750 K; incomplete reaction, and at this point, the absence of Si(l) prevents one from attaining the adiabatic flame temperature of 2930 K. As X/D increases beyond 1.0 wall losses (and radiation losses from particles to the 1800 K walls) begin to rapidly reduce the temperature. Figure 3 shows that at X/D = 0.8 the Si(g) concentration in the region being examined maximizes. The Si condensed phase begins to form at this point and Si(g) is rapidly reduced

FIGURE 2 TEMPERATURE IN SiCl_4/Na MIXING REGION

as the droplets grow via condensation. The calculation was carried out to the point at which about 85% of the available silicon, principally $\text{Si}(g)$ and SiCl_2 , had been condensed. The droplets have rapidly grown, via condensation, to sizes $\approx 0.25 \mu\text{m}$. The mass and number densities ($d m_p / d(\ln r_p)$ and $d n_p / d(\ln r_p)$, respectively) at $X/D = 2.8$, the farthest downstream point at which calculations were pursued, are shown in Fig. 4. From this point on, growth will proceed via agglomeration and will be slow. Roughly speaking, particle mass will double and number density will be halved for $\Delta X/D \approx 3000$, i.e., particle growth is nearly over at $X/D \approx 5$ and the size distributions of Fig. 4 are essentially frozen at this point.

IV. CONCLUSIONS

A model presently being developed¹⁵ to describe the overall silicon collection efficiency of reactors like the Westinghouse prototype has been outlined in this report. The model accounts for the deposition behavior of both silicon vapor and condensed liquid droplets via convection, thermophoresis, Brownian diffusion, and turbulent (eddy) transport of particles. The model is general

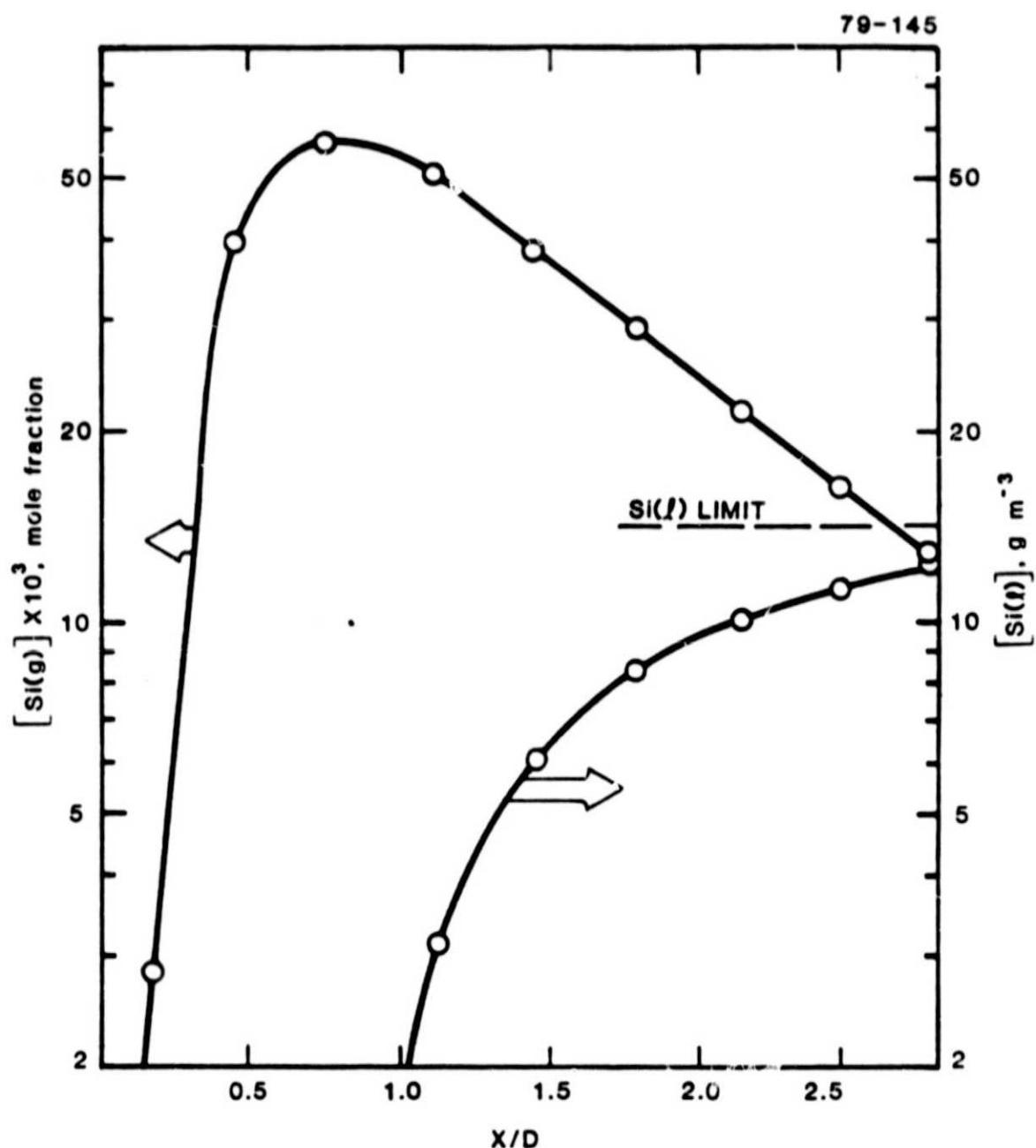


FIGURE 3 Si(g) AND Si(l) CONCENTRATIONS IN SiCl_4/Na MIXING REGION
 $r/d = 0.27$

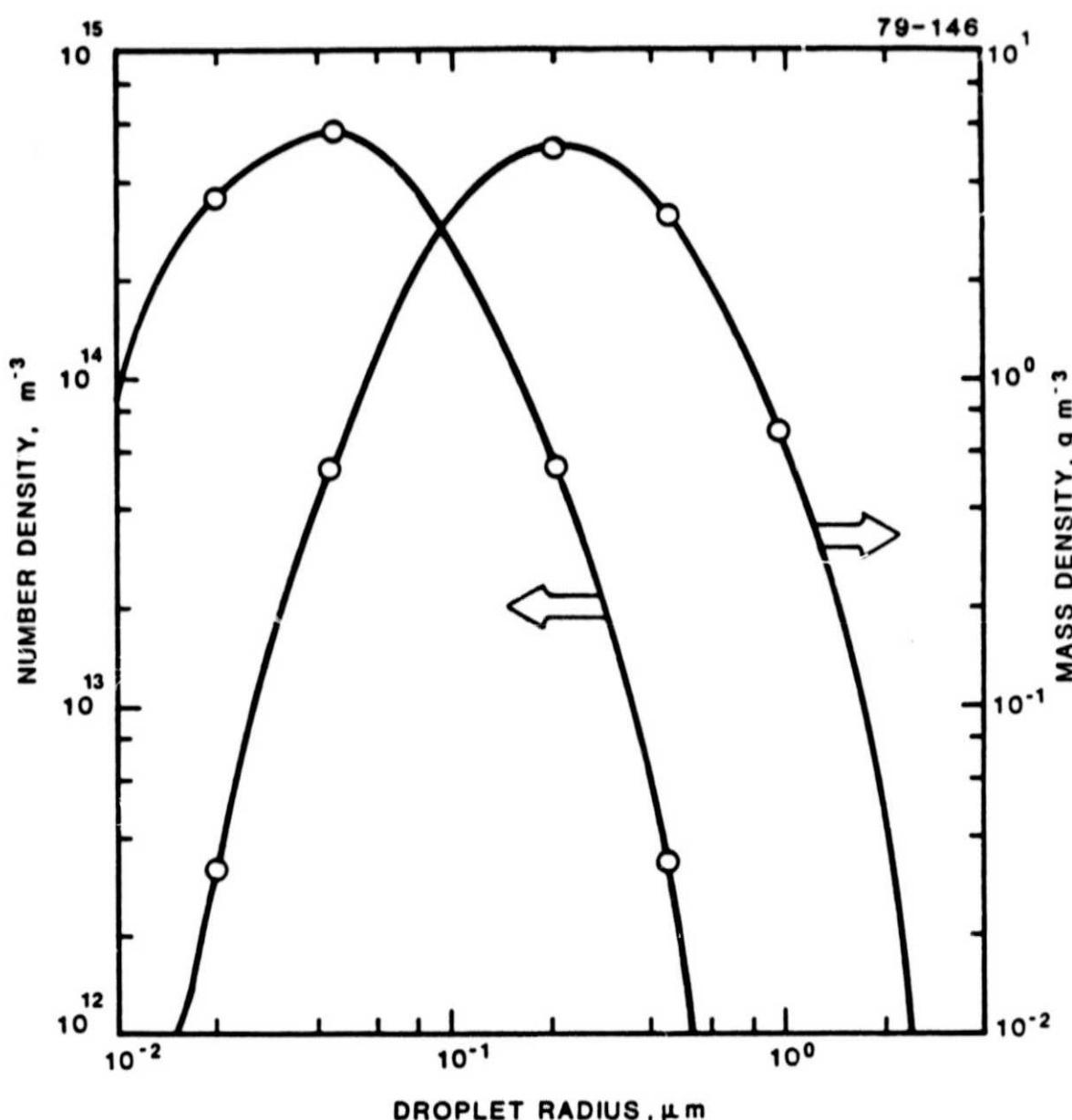


FIGURE 4 SI DROPLET MASS AND NUMBER DENSITIES IN SiCl_4/Na MIXING REGION

$x/d = 2.8$, $r/d = 0.27$

enough to handle the description of a particle size distribution function, provided most of the particle growth has already taken place prior to wall deposition. Unlike existing approaches to this problem,^{10,11} the present model stresses the importance of several novel features that characterize a practical reactor. These include a consideration of (i) variable fluid properties, (ii) a description of the developing, turbulent boundary layers within the reactor, and (iii) particle size distribution effects that invariably falsify deposition behavior,⁹ and hence the reactor's collection efficiency.

In SiCl₄/Na flow reactors in which input enthalpy is insufficient to achieve temperatures above the dew point of silicon, Si(l) droplet formation and growth calculations indicate that droplet sizes will be submicron. Most of the condensed Si in test cases now being examined resides in droplets of 0.2 to 1.0 μm diam.

V. PLANS

During the next quarter, boundary layer calculations will be made to more accurately determine Si collection efficiencies for the Westinghouse reactor, under a variety of operating conditions. The modified LAPP code (CHEMPART) will be used to predict Si droplet size distributions in the Westinghouse and AeroChem reactors so that this information can be used in the abovementioned calculations.

VI. NEW TECHNOLOGY

No reportable items of new technology have been identified.

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